An Automatically Recording Magnetic Balance

著者	HIRONE Tokutaro, MAEDA Seijiro, TSUYA Noboru
journal or publication title	Science reports of the Research Institutes, Tohoku University. Ser. A, Physics, chemistry and metallurgy
volume	6
page range	67-76
year	1954
URL	http://hdl.handle.net/10097/26615

An Automatically Recording Magnetic Balance

Tokutarô HIRONE, Seijirô MAEDA
The Research Institute for Iron, Steel and Other Metals
and Noboru TSUYA
Research Institute of Electrical Communication

(Received November 4, 1953)

Synopsis

A newly designed magnetic balance for plotting automatically the magnetization-temperature curves of the magnetic substances of the ferromagnetic and other types is introduced. The essential part of this instrument consists of the automatic compensensation of the unbalance of the above mentioned balance due to a force acting on the sample. The change of susceptibility due to the rise or fall of the temperature of the specimen can be obtained from the chart in which the electric current for the above-mentioned compensation is recorded. The new apparatus is useful in magnetic analysis of various kinds of magnetic substances.

I. Introduction

If a sample of magnetic substance of paramagnetic or of other type is suspended in a inhomogeneous magnetic field, the force f acting on the sample is given by

$$f = \chi m H \frac{\partial H}{\partial z} ,$$

where χ and m are the susceptibility and the mass of the sample respectively, and $\partial H/\partial z$ is the gradient of the magnetic field in vertical direction z. Several devices(1) with various ideas have been constructed for the measurement of this force. In the present paper, a newly devised instrument capable of recording automatically this force will be introduced.

In the present instrument, the sample is hung from one end of the beam of an analytical balance and from the other end a small magnetic needle is hung through the center hole of a small solenoid. The force acting on the sample is compensated by the mutual force between the magnetic needle and magnetic field produced by the feedback current through the solenoid. The feedback current is produced by a phase sensitive capacity analyzer connected to the pick-up condenser, one plate of which is fixed to the lower end of the pointer of balance. By recording automatically this current the change of magnetic susceptibility due to the temperature change is traced. In this way, the lengthy and tedious work in the magnetic analysis for the physical and metallurgical purposes can be

The 742nd report of the Research Institute for Iron, Steel and Other Metals. This research was supported partly by Grant-in-Aid for Fundamental Scientific Research by the Ministry of Education.

(1) J. D. Eisler, C. R. Newton and W. A. Adcock, Rev. Sci. Instr., **23** (1952), 17. R. F. S. Robertson and P. W. Selwood, Rev. Sci. Instr., **22** (1951), 146. H. M. Allred, Rev. Sci. Instr., **19** (1948), 818. J. W. Clark, Rev. Sci. Instr., **18** (1947). 915. etc.

saved without any loss of accuracy in measurement. The photograph 1 shows the whole arrangement of the present apparatus.

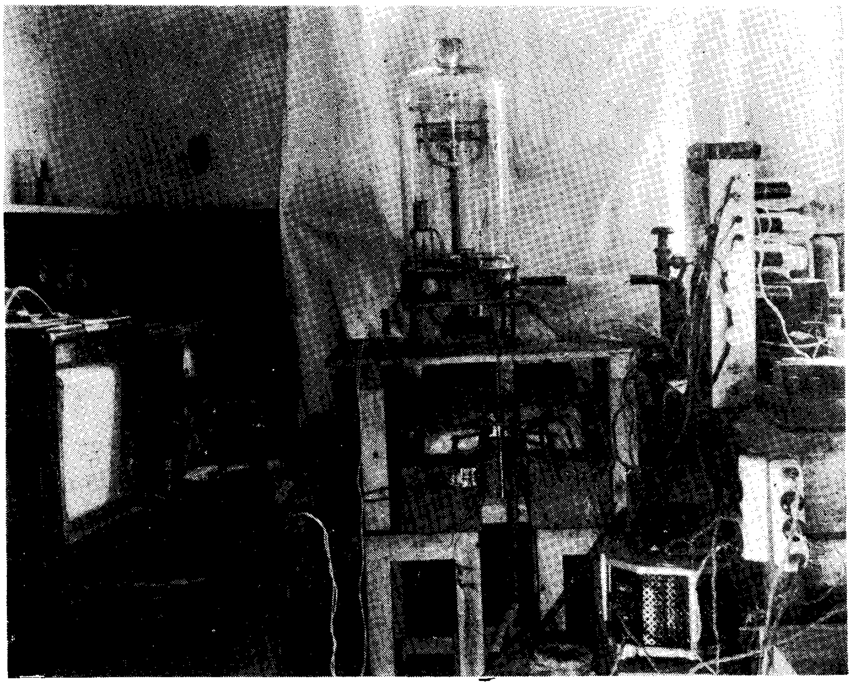

Photo. 1.

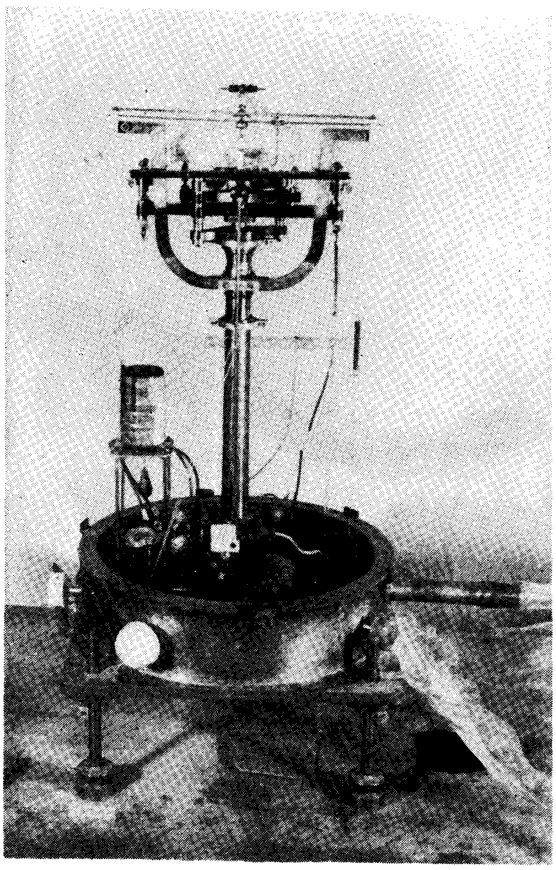

Photo. 2.

II. Magnetic balance

Fig. 1 shows a schematic diagram of the whole apparatus. The appartus consists of two parts, namely, the modified analytical balance or the part of magnetic balance and the compensation circuit for automatic balancing. The whole moving system is enclosed in an evacuated chamber in order to be free from the disturbances due to the buoyancy changes of the sample and other parts, the air flow in the neighbourhood of the balance, and from the condensation of water vapour on the suspension system at low temperatures. Details will be given below. The photograph 2 shows the balance on the large tray, the bell-jar being removed to show the detailed arrangement.

The balance used for this apparatus is an analytical balance of ordinary type modified in the following way: A tray hanging at one end of the beam of balance is replaced by a suspension system consisting of a small needle magnet and a small tray for fractional weights. To the other end a suspension system composed of suspension rod and a small silica cup for a holder of the sample is attached. Under this cup a stretching weight is hung in order to avoid the horizontal shift of the sample due to the magnetic force from the pole pieces of the electromagnet. For the purpose of changing horizontally as well as vertically the position of the rider from the outside of the vacuum chamber, two handles are ground on the side wall of a large brass tray.

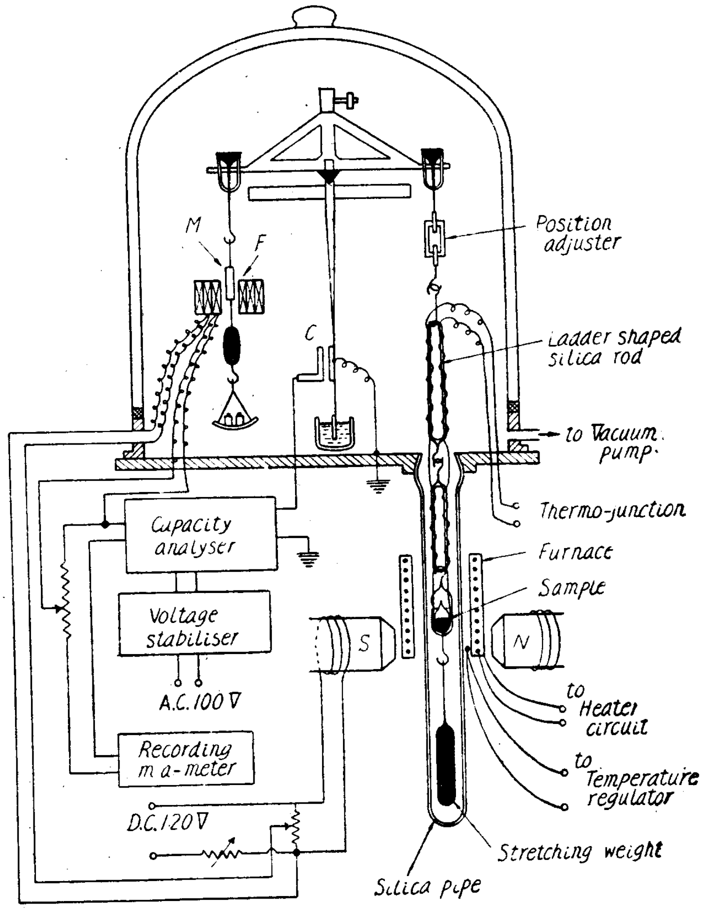

Fig. 1. Schematic diagram of the whole apparatus.

An anti-plate of the pick-up variable condenser C and a vane of the oil damper are fixed with cemedine to the lower end of the pointer of the balance. The above-mentioned magnetic needle is brought to stretch by hunging the lead weight under a tray. The suspension system of the sample is enclosed in a fused silica pipe, whose upper end is ground into a hole of the base plate of the large tray. The important parts of the apparatus will be given separately below.

(i) Pick-up condenser

The moving plate of pick-up condenser is made of mica plate, 3 cm × 4 cm × 0.1 mm, their inner side being covered with a thin foil of tin. As the lead wire of the plate, an enameled copper wire, 0.03 mm in diameter, is soldered by Wood metal, the other end terminating in the base of the large tray. As the lead wire of another fixed plate of the pick-up condenser, a coaxial cable is used, which is connected to the input terminal of the capacity analyzer.

(ii) Sample holder

The sample holder of various types can be used according as the kinds and shapes of samples. For example, as shown in Fig. 2, the sample is placed in the bottom of a cup-shaped holder made of fused silica, 6 mm in diameter, 10 mm in depth and 0.3 mm in thickness, which has two hooks respectively at upper and lower ends. This cup is suspended by the upper hook from the end of the beam through a silica suspension rod which has a length adjuster for the vertical position of the sample, and from the bottom hook a stretching weight made of copper cylinder, 6 mm in diameter and 50 mm in length, is hung by a thin tungsten wire, about 60 mm in length.

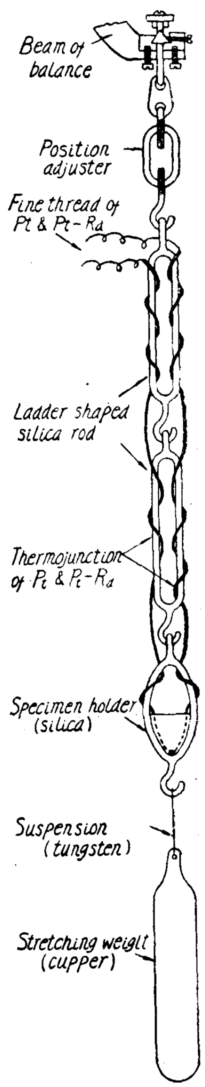

Fig. 2. Schematic diagram of the suspension system.

(iii) Suspension rod

The suspension rod is made up of two pieces of ladder-shaped silica frame, 15 cm in length and 5 mm in width, and has two hooks respectively at upper and lower ends. As a whole system, two sets of such a ladder are used in a way of hook by hook connection in order to simplify the operation of putting in and taking out samples.

(iv) Thermo-junction

For the measurement of the temperature of sample, the Pt-PtRd thermo-junction, 0.1 mm in diameter, is fixed to the inner bottom of the specimen holder so as to be in contact directly with the specimen. The wires of the junction are wound up separately along each rod of the suspension ladders. For perfectly flexible connection of the wires between the suspension system and the balance, the wires of the same kinds, 0.025 mm in diameter, are used. Thereafter, they are taken out through the side wall of the brass tray without disturbing the vaccum. Thus, the thermal contact between thermo-junction and sample is performed sufficiently without any mechanical friction.

(v) Oil damper

As a mechanical damper, a mica foil of 1cm × 2mm is fixed to the lower end of the pointer of the balance and is immersed in diffusion oil filled in a cup. In the present case, it was experimentally confirmed that the change in buoyantic force due to the temperature change of the damper oil and the oscillating fluctuation of the balance beam due to the mechanical disturbances from the laboratory floor were negligibly small.

(vi) Compensation coil and needle magnet

The needle magnet for the compensation of the force produced by the sample is made of alnico magnet, which secures the invariance of its magnetization by the compensation field. The compensation coil is made from three windings on an ebonite bobin having a center hole of 4 mm in diameter. The number of turns of inner coil is about 3,000, which is used for feedback circuit. The middle coil is connected in parallel to the input terminals of the electromagnet in order to compensate the stray magnetic field from the electromagnet in the neighbourhood of the needle magnet. The outer coil is used occasionally as an electric rider by flowing an adequate and constant current during the measurement.

(vii) Vacuum system

The balance system mentioned above is set on a large brass tray in a bell jar which can be evacuated. The tray is made of a brass plate, 1 cm in thickness and 30 cm in width, which is taken as a base plate and on which a brass cylinder, 30 cm in diameter and 7 cm in height, is soldered as a side wall. The bell jar is placed on the flat top of the cylinder wall of the tray, and the vacuum-tight connection between the bell jar and the cylinder wall can be made perfect only by putting a ring-shaped rubber sheet between them. Usually, the measurement may be carried out in a vacuum of the order of 0.01 mmHg, whereas the oil diffusion pump should be used in the case of the susceptibility measurement on a sample which may be oxidized easily or cannot be enclosed in the evacuated sample container.

(viii) Furnace and Dewar vessel

In order to change the temperature of sample, a small electric furnace or a Dewar vessel is inserted from the lower end of the silica tube in which the sample holder is suspended freely from the beam of the balance as mentioned above. The diameter of furnace and Dewar vessel are made as small as possible in order to decrease the gap of the pole pieces of electro-magnet. The furnace is made of silica or porcelain tube, on which the constantan or non-ferromagnetic nichrom wire is wound non-inductively.

III. The electronic circuit for the feedback current

The electronic circuit used in the present apparatus is a phase sensitive capacity analyzer, the principle of which was found by Inoue in 1935. The whole system of electronic circuit is shown in Fig. 3. The circuit is composed of two parts, one being the stabilized power source for plate and heater currents of electronic-tubes and the other being the main circuit containing local oscillator, buffer, phase sensitive capacity ana-

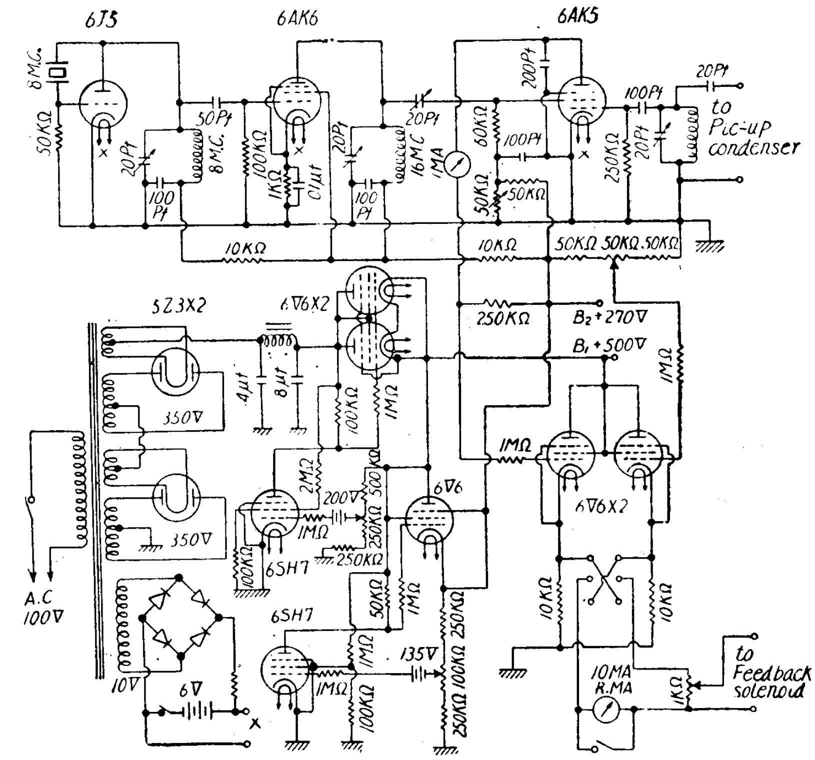

Fig. 3. Circuit diagram of the capacity analyser.

lyzer and cathod follower current amplifier of the balanced type.

Since the circuit of power supply is ordinary one, no detailed explanation will be necessary, but only the principle of capacity analyzer will be explained briefly. A stable high frequency of local oscillator, 16 MC. per second, is fed to the screen grid of 6AK5. The phase of induced high frequency at control grid through a small stray capacity between both grids is different from that at the screen grid. This phase difference takes a large or small value according respectively as the loaded circuit of control grid is inductive or capacitive. Hence, the internal resistance of 6AK5 becomes large or small according respectively as the gap of the pick-up condenser is large or small. Thus, the plate current of 6AK5 is controlled by the change of the capacity of pick-up condenser. This plate current is amplified by 6V6×2 and is fedback to the compensation solenoid. The overall sensitivily of the electronic apparatus is about 5m.a./pico farad.

IV. Measuring procedures

Fig. 1 shows also the principle of measurement. When the sample S is attracted by the magnetic field of electromagnet, the pointer of the balance shifts in a moment and at the same time the capacity of the pick-up condenser decreases and then output current of the capacity analyzer increases. Consequently, the needle magnet M is attracted downwards due to the increase of the current of feedback coil F. By this feedback process, the balancing is recovered instantly. The change of the position of specimen during this process is negligibly small in the usual case, being less than 1 mm even in the largest case. Hence, the change of field acting on the sample due to such a displacement is also negligibly small. The feedback current is recorded by recording milli-ammeter with full scale of 10 m. a., from which susceptibility χ of the sample can be obtained directly through simple calibrations.

The following preliminary adjustments are necessary for the actual measurement of susceptibility.

(i) Compensation of the stray field

The needle magnet M may be attracted due to the weak stray field from the electromagnet and this effect induces the balancing point to take a small displacement. To compensate this effect, an adequate current must be flowed proportionally to the current of electromagnet through the third coil of the compensation solenoid by parallel connection with the circuit of the electromagnet. The compensation by this method is effective in a comparatively wide range of field strength, because the magnetic field produced by the electro-magnet is approximately proportional to the electric current.

(ii) Linearity of the relation between the displacement of the position of sample and the feedback current.

In general, the capacity of the pick-up condenser varies over comparatively wide range and, accordingly, the rate of increase of the compensation current i due to the change of the force f acting on the beam of sample side, di/df, is not constant. By a proper adjustment of the relative position of the stator of pick-up condenser, however, the constant value of di/df can be obtained in a wide range of the displacement of the sample on account of the feedback nature of the circuit. To find this suitable position of the stator, the curve of the force vs. feedback current must be taken for each position of the stator. The sensitivity of measurement can be controlled by adjusting the feedback efficiency of the compensation circuit.

(iii) Determination of the position of sample

The force constant $H\,\partial H/\partial z$ is determined by measuring the force of a certain reference substance of well-known susceptibility, for example, aqueous solution of $NiCl_2$. The position of the sample is successively displaced along the vertical line passing through the center of the gap between the pole pieces of the electromagnet. In this way, the force constant can be determined as a function of position of the sample, as shown in Fig. 4. From this result, the suitable position to be occupied by the sample will be found. It was confirmed that the change in $H\,\partial H/\partial z$ due to that of the position of sample could be made negligibly small by taking an appropriate position of the sample. Usually, it is slightly lower than that corresponding to the maximum of force.

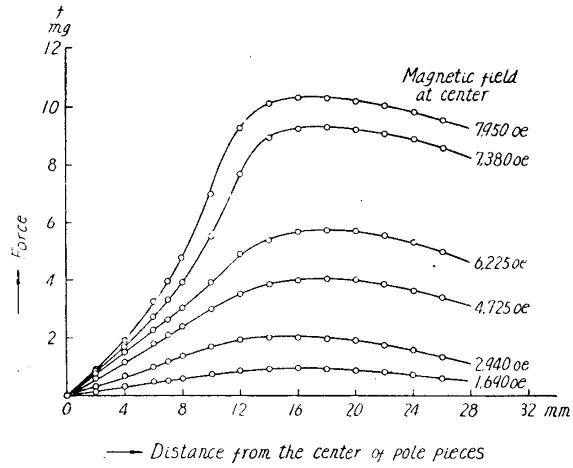

Fig. 4. Curves of the force vs distance from center of pole pieces of the electromagnet.

(iv) Empty test

By closing the circuit of the electromagnet a steady deviation of the feedback current can be observed, even the sample is not placed, owing to the following two causes: (a) the para-or dia-magnetic nature of the parts of suspension system (the sample container, silica rods, Pt-PtRd thermo-junction and stretching weight made of copper), and (b) the change in the capacity between the electromagnet and the circuit of pick-up condenser. The former effect can be removed by adjusting the position of copper weight. The latter gives a constant deflection and can also be removed perfectly by shielding the circuit.

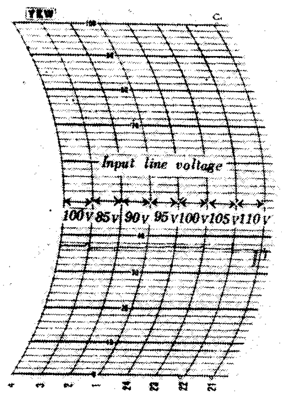

Fig. 5. The influence in the recorded chart of feedback current due to the change of the input voltage of a.c.-line.

(v) The effect of the fluctuation in a.c.-line voltage

The troubles due to the fluctuation of a.c.-line voltage is almost negligibly small due to the characteristics of the feedback system of the apparatus. In the present case, a precise stabilization for plate voltage supply is performed by using an electronic stabilizer. For the source of filament current the stabilizer of floating battery type with a selenium rectifier is used. The relation of the recorded current versus a.c.-line voltage is shown in Fig. 5. As shown in this figure, when the line voltage fluctuation is less than ± 3V, the change in feedback current corresponds to about ± 0.04 mg and, hence, the effect of voltage fluctuation is negligibly small in the usual case.

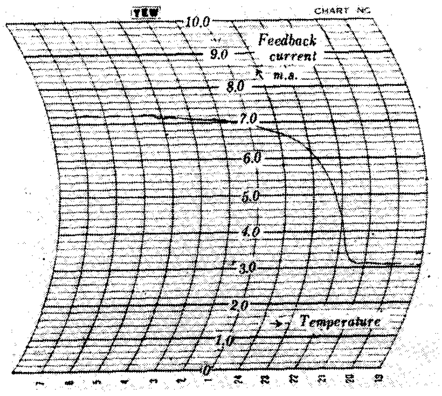

Fig. 6. Recorded chart of the change of saturation magnetization of Ni due to temperature rise.

(vi) The shield of the electronic circuit

The circuit used in the present apparatus has a very high sensitivity to the change of capacity and, accordingly, the shield of the lead wire must be so highly guaranteed as to make the effect on the feedback current negligibly small even if the hand or the body of the operator approaches the apparatus.

As an example of the measurement of the curve of susceptibility versus temperature, the recorded chart in the case of nickel is shown in Fig. 6.

V. Discussion on thc accuracy

Lastly, the accuracy of measurement by the present apparatus will be discussed. The errors due to (1) the stray field from the electromagnet, (2) the magnetic behaviours of the suspension system, (3) the effect of the stray capacity and (4) fluctuation of a.c.-line voltage must first be considered. These errors could be made to negligibly as small as those mentioned in Sect. IV. The error to be considered next is that in the linearity of the relation between the feedback current and the force. Such a linearity will be calibrated by the discontinuous displacement of the rider. Recorded chart in the case of such calibration is shown in Fig. 7. The figure shows the change of feedback current due to that of the force. A slight change in counter balance due to the change of mass of each sample causes a slight change in linearity. This effect was less than 0.05 mg at most. The mechanical disturbauces will make the fluctuations of the recorded curves, which are usually less than 0.02 mg and, therefore, this effect causes no significant error. The most important errors may arise from the following sources, which are common troubles in the measurements by any magnetic balance: (i) The inhomogenuity of the magnetic field in the specimen; (ii) the error in manufacturing or treating

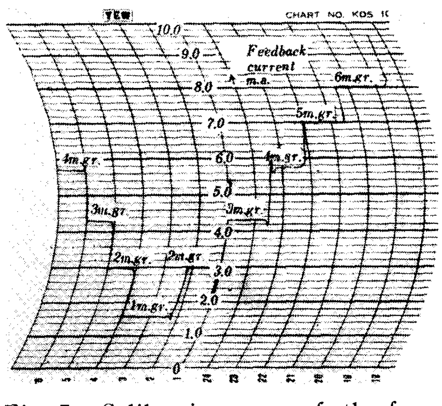

Fig. 7. Calibration curve of the force vs feedback current

the reference sample; (iii) the ambiguity in the position and the shape of sample (iv) the difference in volume between the reference sample and the sample to be measured. The error (i) is inevitable except the case in which a very large electromagnet is used. In the present case, the field gradient along the vertical direction was usually less than 40 Oe/mm. The error (ii) corresponds to that of chemical analysis of the reference sample. The error (iii) is significant, especially in the case of measurements with the powdered sample, in which vacuum must be kept tight in the sample container made of fused silica or glass. This effect also is usually inevitable owing to the difficulty of making the sample container with precise dimension. In the present case, the fluctuation of the size of sample container was less than about 1 mm. For a very accurate measurement, very thin and small capsule with a shape of perfect ellipsoid must be used. To minimize the error (iv) the volume of reference sample must be made as equal as possible to that of the sample to be measured. When the susceptibility of the sample is very large, we used sometimes metallic nickel at a temperature above its Curie point as the reference sample.

From the above discussions, it is concluded that there are some troubles for choice of the most suitable reference sample. For this reason, we propose here a method for absolute measurements of susceptibility without reference substances by using a standard coil instead of an electromagnet. In such a case, the distribution of the magnetizing field is exactly computable from the size of the standard coil.

VI. Characteristics of the present apparatus

The characteristic points of the present magnetic balance are as follows:

(i) The operation of temperature change of the sample is very easily performed in the range from the temperature of liquid He to about 1,000°C only by the replacement of a small furnace and Dewar vessel.

(ii) For the exchange operation of the sample, only the readjustment of the counter weight according to the mass difference of the sample is required and no other adjustment. The total time required from the exchange operation of the samples to the start of the subsequent measurement is less than about 10 minutes.

(iii) The adjustment and calibration of measurement are comparatively simple. The stability of the whole apparatus is so good that no readjustment of the main parts is necessary even after several days. Therefore, the calibration is not required for each measurement except in the case of an especially accurate measurement.

(iv) The mechanical disturbance for a short period, such as the effect of the operation of the rotary pump or rough walking in the neighbourhood of the apparatus, has no influence on the measurement; on the contrary, the mechanical disturbance of comparable period with the balance itself slightly affected the recorded chart. For example, the effect of putting on or off the furnace was hardly detectable. In the present apparatus, there is utterly nothing of difficulties or troubles arising from the optical system which is usually used in automatic recording system of the magnetic balance.

(v) The sensitivity can be brought to sufficiently high order for a wide range of the mass of sample. In an ordinary case, the sensitivity is of the order of 1 mg per m. a. and can be adjusted by changing the order of feedback efficiency. In the case of the short time measurement, the sensitivity brought to an extremly high value by inversing the sign of feedback, that is, from the negative feedback to the positive one, although the stability decreases not so strongly. Fig. 8 shows such relations between the force and the output current of the capacity analyzer as the parameter of the degree of feedback. In the actual case, the shadowed range in these curves may be used for the compensation current.

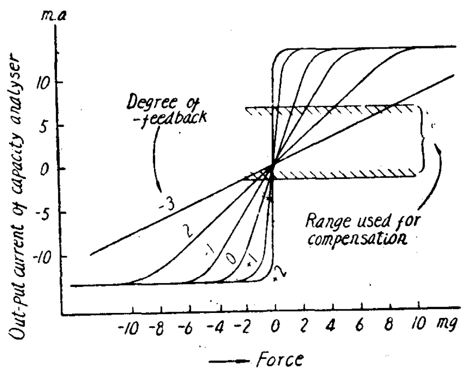

Fig. 8. Relations between the force and the out-put current of the capacity analyser.